PATENT SPECIFICATION

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698,127

Date of filing Complete Specification Feb. 4, 1952.

Application Date March 14, 1951. No. 6 29/51.

Complete Specification Published Oct. 7, 1953.

Index at acceptance :—Class 2(iii), C1c.

COMPLETE SPECIFICATION

Improvements in or relating to the Production of Tetrafluorodichloroethane

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, London, S.W.1, a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to an improved process for the production of 1,1,2,2-tetrafluoro-1,2-dichloroethane.

The additive reaction of chlorine with tetrafluoroethylene would provide an attractive route to the production of 1,1,2,2-tetrafluoro-1,2-dichloroethane, if the reaction was easy to carry out and if sufficiently high yields of the reaction product were obtained. However, when chlorine is contacted with tetrafluoroethylene in the dark in the presence of a diluent such as carbon tetrachloride at temperatures of from 0° C. to 20° C. no reaction, or only a very slow reaction ensues. Again if the reactants are contacted in the vapour phase in the absence of a diluent, particularly, at elevated temperatures, reaction is liable to occur with explosive violence.

According to the present invention a process for the production of 1,1,2,2-tetrafluoro-1,2-dichloroethane comprises reacting chlorine and tetrafluoroethylene in the presence of an organic liquid diluent which is substantially unreactive towards the reactants and reaction product, while exposing the system to actinic radiation.

In the present invention, a particularly useful liquid diluent is carbon tetrachloride, which material is capable of dissolving therein small amounts of chlorine and tetrafluoroethylene. When employing carbon tetrachloride good results may be obtained when the reaction is carried out at a temperature in the range 0° C. to 50° C. A reaction temperature in the approximate range 20° C to 35° C. suitably 30° C., is particularly preferred since at this temperature the reaction product continually distils over.

A suitable manner of carrying out the invention in a continuous manner is to pass tetrafluoroethylene and chlorine, either of which may be in slight molar excess over the other reactant, into a vessel containing carbon tetrachloride, the system being exposed to actinic radiation. An inert gas such as nitrogen may also be passed in the free space above the carbon tetrachloride diluent. A reaction temperature of 30° C. is employed and the reaction product which continually distils over is collected in a low temperature trap and is then fractionated.

The following Example illustrates but does not limit the invention.

EXAMPLE

A mercury vapour lamp emitting light of various wave-lengths including those in the range 3600 to 4400Å was situated as near as could be conveniently arranged (of the order of 3 inches) from a glass vessel equipped with a stirrer and containing carbon tetrachloride. 337 parts of chlorine and 520 parts of tetrafluoroethylene were passed slowly into the liquid and a slow stream of nitrogen was passed into the free space above the carbon tetrachloride.

A reaction temperature of 30 to 34° C. was maintained. The reaction product which distilled over was collected in a low temperature trap, fractionated and found to be almost wholly 1,1,2,2-tetrafluoro-1,2-dichloroethane.

773 parts of the pure reaction product was obtained, a yield of 95% based on chlorine being realised.

What we claim is:—

(1) A process for the production of 1,1,2,2-tetrafluoro-1,2-dichloroethane which comprises reacting chlorine and tetra-

fluoroethylene in the presence of an organic liquid diluent which is substantially unreactive towards the reactants and reaction product while exposing the system to actinic radiation.

(2). A process according to Claim 1 in which the liquid diluent is carbon tetrachloride and in which a reaction temperature in the range 0° to 50° C. is employed.

(3). A process according to Claim 2 in which a reaction temperature in the range 20° C. to 35° C., suitably 30° C., is employed.

(4). A process according to any of the preceding claims in which the reaction is carried out in the presence of an inert gas, such as nitrogen.

(5). A process for the production of 1,1,2,2-tetrafluoro-1,2-dichloroethane substantially as hereinbefore defined with reference to the Example.

(6). 1,1,2,2 - tetrafluoro - 1,2 - dichloroethane whenever produced by a process according to any of the preceding claims.

ALFRED O. BALL,
Agent for the Applicants.

PROVISIONAL SPECIFICATION

Improvements in or relating to the Production of Tetrafluorodichloroethane

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, London, S.W.1, a British Company, do hereby declare this invention to be described in the following statement:—

This invention relates to an improved process for the production of 1,1,2,2-tetrafluoro-1,2-dichloroethane.

The additive reaction of chlorine with tetrafluoroethylene would provide an attractive route to the production of 1,1,2,2-tetrafluoro-1,2-dichloroethane, if the reaction was easy to carry out and if sufficiently high yields of the reaction product were obtained. However when chlorine is contacted with tetrafluoroethylene in the dark in the presence of a diluent such as carbon tetrachloride at temperatures of from 0° C. to 20° C., no reaction ensues. Again if the reactants are contacted in the vapour phase in the absence of a diluent, particularly, at elevated temperatures, reaction is liable to occur with explosive violence.

We have now found that exceptionally high yields of 1,1,2,2-tetrafluoro-1,2-dichloroethane may be obtained by reacting chlorine and tetrafluoroethylene in the presence of a diluent while exposing the system to actinic irradiation.

A liquid diluent which is substantially unreactive towards chlorine and tetrafluoroethylene, such as carbon tetrachloride, is particularly useful for the purpose of the present invention. The reaction temperature may be varied over a wide range, depending on the particular liquid diluent employed. For example, when carbon tetrachloride is employed as diluent, good results may be obtained at temperatures in the range 0°C. to 50° C.

A suitable manner of carrying out the invention in a continuous manner is to pass tetrafluoroethylene and chlorine, if desired in slight molar excess, into a vessel containing carbon tetrachloride, the system being exposed to actinic irradiation. An inert gas such as nitrogen may also be passed in the free space above the carbon tetrachloride diluent. The reaction temperature is maintained, suitably at 30° C., and the desired reaction product continuously distils over. The distillate is collected in a low temperature trap and is then fractionated.

The following Example illustrates but does not limit the invention.

EXAMPLE

A mercury vapour lamp emitting light of various wave-lengths including those in the range 3600 to 4400Å was situated as near as could be conveniently arranged (of the order of 3 inches) from a glass vessel equipped with a stirrer and containing carbon tetrachloride. 337 parts of chlorine and 520 parts of tetrafluoroethylene were passed slowly into the liquid and a slow stream of nitrogen was passed into the free space above the carbon tetrachloride.

A reaction temperature of 30 to 34° C. was maintained. The reaction product which distilled over was collected in a low temperature trap, fractionated and found to be almost wholly 1,1,2,2-tetrafluoro-1,2-dichloroethane.

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Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press.—1953.
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